

## MECHANICAL STABILIZATION OF GRAPHENE AEROGELS BY VULCANIZATION WITH PURE SULPHUR

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Graphene aerogels (see Figure 1) are very useful materials for those technological applications where high-surface development and good electrical conduction characteristics are required [1]. Such nanostructured materials can be prepared by drying a high concentrated graphite-nanoplatelets (GNP) colloidal suspension, which is obtained by treating expanded graphite with ultrasounds in a liquid medium (e.g., octane). However, the resulting mechanical properties are not enough for technological applications.

In order to give more strength to this material, it was vulcanized with pure sulphur (at 180°C). The carbon-sulphur chemical reaction can be advantageously used for the mechanical stabilization of the very fragile spongy graphite material [2]. The introduction of sulphur in the graphite structure is quite simple since the sulphur molecules ( $S_8$ ) are quite soluble into non-polar organic media and it can be dissolved in the GNP colloid before the drying process.

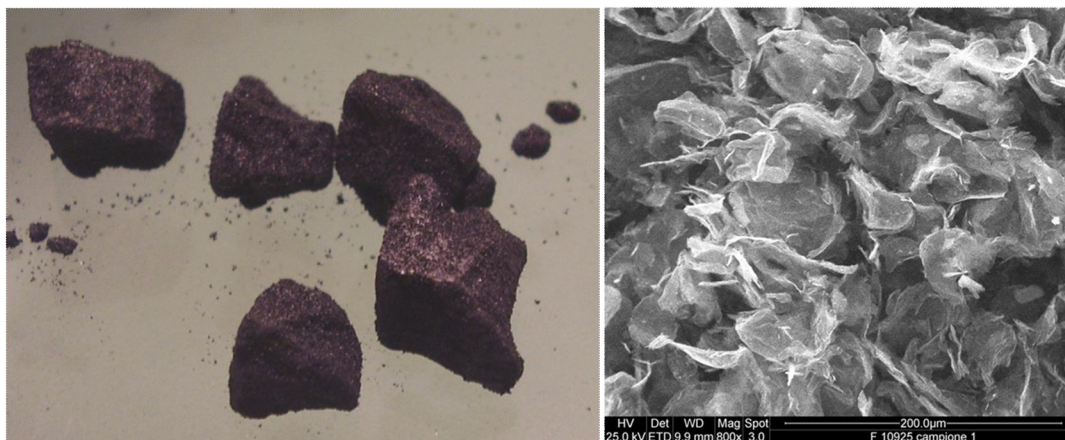
In particular, graphene is a very good substrate for chemical functionalization, since its reactivity is comparable with that of other polycyclic aromatic hydrocarbons. The presence of carbon-carbon double bonds (C=C) makes possible also radical addition reactions. Sulphur molecules ( $S_8$ ) decompose in the molten form, producing linear bi-radicals  $c-S_8 \rightarrow \bullet l-S_8 \bullet$  ( $\lambda$ -transition) [3], which are able to cross-link the graphene-based framework constituting the aerogel.

Such a process significantly improves the material mechanical stability (see Figure 2). To verify the presence of S in the vulcanized material and to establish its distribution in the sample composition of our materials, we carried out energy dispersive spectroscopic (EDS) mapping. EDS combined with scanning electron microscopy (SEM) images of the graphene-sulphur composite (after the vulcanization process) showed the presence of such sulphur phase segregated at the edges of graphene sheets. In addition, differential scanning calorimetry (DSC) was used as a really convenient approach to study this vulcanization process, in fact the complete cross-linking of the graphene structure was evidenced by the disappearance of the pure sulphur phase melting (see Figure 3). The amount of residual sulphur in the vulcanized material was evaluated by thermo-gravimetric analysis (TGA) and it corresponded to ca. 30% by weight. Further characterization approaches used to investigate the morphology and the structure of graphene aerogels have been: large angle X-ray powder diffraction, Fourier-transform spectroscopy (FT-IR), X-ray fluorescence spectroscopy.

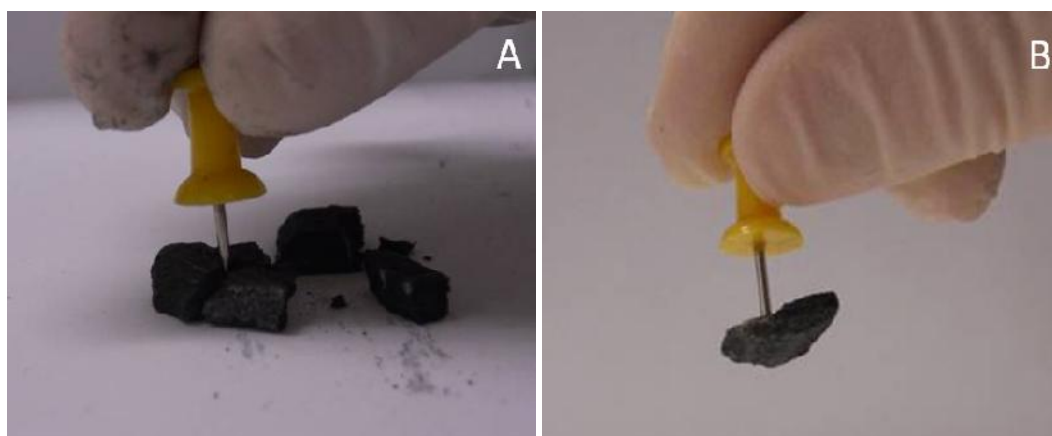
### References

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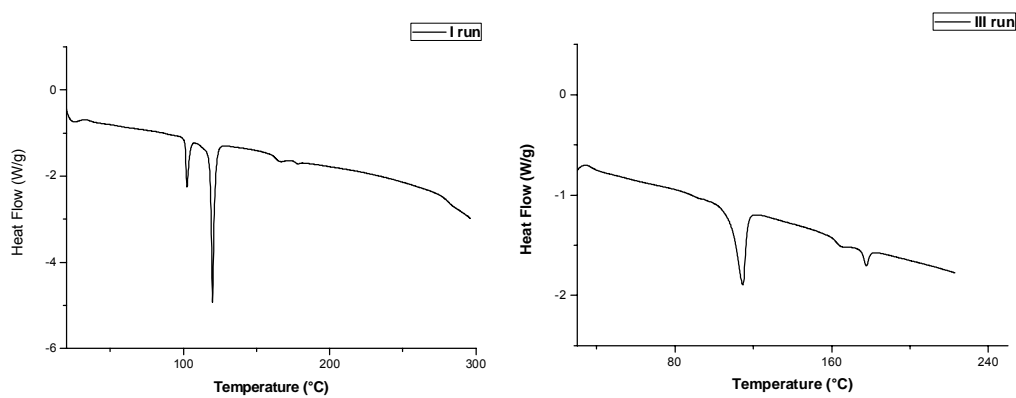
## Figures



**Figure 1** - Graphene aerogel samples (left-side) and SEM-micrograph (right-side).



**Figure 2** - Graphene aerogel samples before (A) and after (B) the vulcanization treatment



**Figure 3** – DSC-thermogram of the reactive sulphur/GNPs mixture